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**A method of manufacturing syntactlc foam**

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## ABSTRACT

A method of manufacturing syntactic foam is disclosed which includes the steps of combining a polymer, microspheres and a solvent to form a  
5 slurry. At least a portion of the solvent is removed through a porous wick, and conditions are applied which substantially solidify the polymer.

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THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method of forming a syntactic foam including the steps of:
  - (a) combining a polymer, microspheres and a solvent to form a slurry;
  - (b) thereafter removing a portion of said solvent through a porous wick;
  - 5 and
  - (c) applying conditions which substantially solidify said polymer.
2. The method of claim 1 wherein said polymer and microspheres are combined to form a paste, and thereafter the solvent is combined with said paste to form a slurry.
- 10 3. The method of either claim 1 or 2 wherein said microspheres, said polymer and said solvent are combined together in incremental portions.
4. The method of any preceding claim wherein the slurry is inserted in a mould having a desired physical shape, and the solvent is removed from said slurry while the slurry is in a mould to form a syntactic foam with the desired
- 15 physical shape.
5. The method of claim 4 wherein said mould includes an inner surface in contact with said slurry, and said inner surface forms at least part of the porous wick.
6. The method of any preceding claim wherein said porous wick is
- 20 selected from plaster, clay, ceramic, cement, concrete, finely perforated plastic sheets with absorbent backing, finely perforated metallic sheets with absorbent backing, fine metallic mesh/gauze with absorbent backing, rigid porous foams or sponge-like material.
7. The method of any preceding claim wherein said solvent is water or a
- 25 volatile alcohol or a ketone.
8. The method of any preceding claim wherein said solvent is selected from water, butyl alcohol, isopropyl alcohol, ethyl alcohol, methyl alcohol, acetone or mixtures thereof.
9. The method of claim 8 wherein said solvent is acetone.
- 30 10. The method of any preceding claim wherein said polymer is a resin based compound.
11. The method of claim 10 wherein said resin based compound is selected from amino resins, epoxide resins, phenolic resins, tar acid resins,

26. The method of any one of claims 20 to 25 wherein the syntactic foam has total void spaces formed from interstitial voids between the microspheres, and microsphere void spaces within the microspheres, wherein the weight ratio of microspheres to polymer forms a syntactic foam with a total interstitial void space greater than the total microsphere void space.
27. The method of any preceding claim wherein the syntactic foam has a density of between 0.05 and 0.5 grams/cm<sup>3</sup>.
28. The method of claim 27 wherein the density of the syntactic foam is between 0.055 grams/cm<sup>3</sup> and 0.15 grams/cm<sup>3</sup>.
29. A syntactic foam when made by a method including the steps of:
- (a) combining a polymer, microspheres and a solvent to form a slurry;
  - (b) thereafter removing a portion of said solvent through a porous wick; and
  - (c) applying conditions which substantially solidify said polymer.
30. A syntactic foam wherein the syntactic foam has total void spaces formed from interstitial voids between the microspheres, and microsphere void spaces within the microspheres, and wherein the total interstitial void space is greater than the total microsphere void space.
31. A syntactic foam formed by a method including the steps of:
- (a) combining a polymer, microspheres and a solvent to form a slurry;
  - (b) thereafter removing a portion of said solvent through a porous wick; and
  - (c) applying conditions which substantially solidify said polymer;
- wherein the syntactic foam has total void spaces formed from interstitial voids between the microspheres, and microsphere void spaces within the microspheres, and wherein the total interstitial void space is greater than the total microsphere void space.
32. A syntactic foam wherein the density of the syntactic foam is between 0.055 grams/cm<sup>3</sup> and 0.15 grams/cm<sup>3</sup>.
33. A method of forming syntactic foam substantially as herein described with reference to any one of the examples.

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Complete Specification for the invention entitled:

**"A METHOD OF MANUFACTURING SYNTACTIC FOAM"**

Our Ref: **IRN 644841**

The following statement is a full description of this invention, including the best method of performing it known to applicant:

## **A METHOD OF MANUFACTURING SYNTACTIC FOAM**

### **Field of the Invention**

The present invention relates generally to syntactic foams and more particularly to an improved method of manufacturing syntactic foam.

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### **Background of the Invention**

Syntactic foams are composite materials whose resinous matrix is embedded with preformed particles such as glass or ceramic microspheres. Syntactic foams distinguish themselves from other foams by the fact that hollow or solid spheres of a predetermined size and packing composition are used to control the density of the foam.

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Syntactic foams have been used for purposes which require a low density packing material such as undersea/marine equipment for deep-ocean current-metering, anti-submarine warfare, sandwich composites, the aerospace industry and the automotive industry.

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Difficulties have been experienced, however, in producing syntactic foams that have a density which is comparable to conventional foams. Typical densities of syntactic foams vary between 0.3 and 0.5 g/cm<sup>3</sup>, whilst conventional foams typically vary between 0.01 and 0.1 g/cm<sup>3</sup>. The density of syntactic foams has generally been restricted by the limited porosity of the foams. Porosity is a measure of the total void volume of the syntactic foam, and constitutes the sum of the void volume of the microspheres and the interstitial void volume. Using current methods of syntactic foam manufacture, the void volume provided by the microspheres is greater than the void volume provided by the interstitial spaces. Thus, the density of syntactic foams have been limited by the void volume of the microspheres. As such, the application of syntactic foams have been limited.

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### **Summary of the Invention**

An aim of the present invention is to provide a novel method of manufacturing syntactic foam which is ideally suitable for manufacture of low density foams and which is simple and inexpensive.

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Accordingly, in a first aspect of the invention, there is provided a method of forming a syntactic foam including the steps of:

- a) combining a polymer, microspheres and a solvent to form a slurry;

- b) thereafter removing at least a portion of the solvent through a porous wick; and
- c) applying conditions which substantially solidify the polymer.

The applicant has found that by manufacturing syntactic foams in accordance with the method of the invention, it is possible to provide adequate coating of the microspheres to form the syntactic foam using a high microsphere mass fraction. This occurs primarily by the solvent reducing the viscosity of the polymer thereby allowing less polymer to be used to coat the microspheres and by causing at least a portion of the solvent to be removed from the composition. The applicant has found that using this method the porosity of the interstitial void volume of the syntactic foams can be greater than the void volume of the microspheres. This enables syntactic foam densities of less than  $0.15 \text{ g/cm}^3$  to be produced. Further, the technique is simple and is ideally suited for mass production and easily applicable for manufacturing large items.

In a preferred form, the method further includes the steps of combining the polymer and the microspheres to form a paste, and thereafter adding the solvent to the paste to form the slurry and mixing the slurry to achieve dispersion of the microspheres prior to removal of the solvent. Even more preferred is that a final portion of microspheres necessary to maintain the desired mass fraction is added alternatively with the solvent. This helps to maintain a consistency which allows easy pouring.

In a preferred aspect of the invention, the solvent is removed from the slurry while the slurry is in a mould such that the syntactic foam forms a desired physical conformation upon removal of the solvent. In an especially preferred aspect of the invention, the syntactic foam is made by a method in which the mould is the porous wick for removal of the solvent. The porous wick itself may be made from any material that is capable of soaking up or removing the solvent. The most preferred porous wick is made of plaster. Alternatively, the porous wick may be made of clay, ceramic, cement, concrete, finely perforated plastic or metallic sheets with absorbent backing, fine metallic mesh/gauze with absorbent backing, rigid porous foams or sponge-like material.

An advantage of this arrangement is that the removal of the solvent through the mould walls does not cause any major disturbance of the distribution of the microspheres. As such, the resulting syntactic foam is able to maintain an even density and tactile strength.

5 To allow for ease of moulding and speed of manufacture, the amount of solvent added is sufficient to form a slip by maintaining a soupy consistency. The mixture can then be quickly poured into the desired mould.

It is a preferred aspect of the present invention that the solvent is a volatile alcohol or ketone. The most preferred alcohols are butyl alcohol, isopropyl alcohol, ethyl alcohol or methyl alcohol. Even more preferred is that  
10 the solvent is the ketone acetone. Alternatively, the solvent may be water or a mixture of water and alcohol or water and acetone. In general, a slip made using water requires a lower ratio of solvent to polymer and hardener. The volume ratio of water to polymer is generally between 9:1 and 6:1. The  
15 volume ratio of acetone to polymer is between 40:1 and 9:1. The volume ratio of water to polymer and acetone to polymer varies with the ratio of microspheres to polymer. The greater the ratio of microspheres to polymer, the greater the volume of solvent required to maintain a consistency necessary to allow pouring of the mixture. Ideally, the volume ratio of solvent  
20 to polymer is sufficient to maintain a slip.

The polymer may be dissolved in the solvent, or dispersed throughout the solvent as a suspension. In a preferred aspect of the invention, the polymer is dissolved in the solvent. It is especially preferred that the polymer is dissolved in acetone. Alternatively, the polymer is homogenised or  
25 dispersed throughout the solvent as an insoluble suspension.

The polymer used in the method of making the syntactic foam of the present invention may be any resin that is effectively employed in syntactic foam manufacture. The classes of resins used for syntactic foam manufacture may include but is not restricted to amino resins, epoxide resins,  
30 phenolic and other tar acid resins, urea and melamine resins, vinyl resins, styrene resins, alkyd resins, acrylic resins, polyethylene resins, polypropylene resins, petroleum polymer resins, polyamide resins, polycarbonate resins, acetal resins, flourohydrocarbon resins, polyester



resins and polyurethane resins. Further, the polymer may be water soluble copolymers such as poly(N-vinylpyrrolidone-vinyl acetate). A particularly preferred resin is a phenolic resin.

Depending on the nature of the polymer, the polymer may include a crosslinking agent or hardener which causes solidification of the polymer material. In cases where the polymer includes a cross-linker to solidify, it is preferred that the weight ratio of hardener to polymer is 1:10. However, it is envisaged that the ratio of hardener to polymer will vary depending on the type of polymer and the degree of crosslinking that is desired. For example, increased amounts of hardener will allow low temperature curing and will speed up gelation. Decreasing the relative amounts of hardener to polymer to a weight ratio as low as 5:100 may be possible, but higher curing temperatures and longer gelation times may be required.

Preferably, the syntactic foam of the present invention is a low density syntactic foam. To reduce the density of the syntactic foam, the microspheres are advantageously hollow gas filled spheres. They are generally made of soda-lime-borosilicate glass, but may also be made from, but not limited to, any one of the following types: carbon, ceramic, phenolic, epoxy or melamine-formaldehyde. For some applications it is envisaged that the microspheres used in the method of the present invention will be thermoplastic. The microspheres may be untreated or precoated. Preferably, the surface of the microspheres is precoated with a coupling agent, such as a silane coupling agent. The microspheres may also be dried prior to use. It is also envisaged that the syntactic foam of the present invention may also comprise a non homogenous mixture of microspheres made of different compounds.

As indicated above, the porosity of the syntactic foam is a combination of the void volume of the microspheres and the interstitial void volume between the microspheres. An advantage of the preferred form of the invention is that the method allows for high interstitial void volume to be produced because adequate coating of the microspheres can occur using low amounts of polymer. The method also does not induce forces in the mix that results in increased packing of the microspheres. As such, it is possible

using the method of the invention to lower the density of the syntactic foam by creating a high interstitial void volume. The applicant has found that the density of the syntactic foam produced by the method of the present invention is lowest when the interstitial void volume is greater than the total  
5 void volume of the microspheres. Thus, the greater proportion of porosity in the low density syntactic foam of the present invention results from the interstitial voids between the microspheres.

In a preferred aspect of the invention, the microspheres are less than 420 micrometres in diameter. More preferably they are less than 300  
10 micrometres. Even more preferably they are less than 150 micrometers in diameter. To vary the density of the syntactic foam and to obtain tighter packing of the microspheres, the method of making syntactic foam can use microspheres of different sizes. In a preferred aspect of the invention, small  
15 microspheres are mixed with large microspheres to produce a closely packed syntactic foam with a high microsphere volume fraction.

The density and mechanical strength of the syntactic foam can be controlled by the weight-ratio of microspheres to polymer. Depending on the structural properties required, the weight ratio of microspheres to polymer is typically between 8:2 and 2:8. It is preferred that the weight ratio of  
20 microspheres to polymer is between 8:2 and 1:1. A more preferable weight ratio of microspheres to polymer is between 8:2 and 6:4. Still even more preferred is a weight ratio of microspheres to polymer of between 8:2 and 7:3. However, weight ratios of microspheres to polymer of between 1:1 and 2:8 are also effective in producing a syntactic foam. Lower ratios of  
25 microspheres to polymer results in a denser foam with greater mechanical strength. In the case of foams of greater mechanical strength, weight ratios of between 4:6 and 2:8 are preferred. For greater mechanical strength, weight ratios of between 3:7 and 2:8 are preferred. It is likely that the effective ratio of microspheres to polymer will vary with the properties of the polymer and  
30 the microspheres employed. Accordingly, polymers with greater wettability may be effective at weight ratios of greater than 8:2.

Preferably, the density of the syntactic foam produced by the method of the present invention is between 0.05 and 0.5 g/cm<sup>3</sup>. Even more

preferably, the density of the syntactic foam is between 0.055 g/cm<sup>3</sup> and 0.15 g/cm<sup>3</sup>.

5 The polymer and microspheres may be mixed by any method which provides sufficient dispersion of the microspheres through the polymer but does not damage a proportion of the microspheres. The method of mixing the polymer/microsphere mixture with the solvent may be any method which results in dispersion of the microspheres throughout the mixture. It should be noted that the method of mixing the microspheres and polymer need not be the same as the method of mixing the polymer/microsphere mix with the solvent. It is however preferred due to ease of manufacture that the polymer and microspheres are mixed in the same manner as the solvent and polymer/microspheres.

10 Following removal of the solvent through the porous wick, the syntactic foam of the present invention may be treated under conditions to cure or solidify the polymer. These may be conditions such as elevated or lowered temperature, stillness, catalytic activity, or any other conditions which allow solidification of the polymer. The conditions will vary with the type of polymer used. The preferred condition is temperature. The temperature used to cure the polymer may be any temperature which does not damage the polymer or the microspheres. The temperature will vary depending on the type of polymer used and the nature of the microspheres or the solvent. Preferably, the temperature will be between 0° and 60°C. Even more preferably, the temperature will be between 20° and 35°C. Prior to heating, the majority of the solvent is preferably removed through/by the porous wick. Thus, heating should be only sufficient to allow curing of the resin. This will reduce damage to both the resin and the microspheres.

25 In yet a further aspect, the present invention relates to a syntactic foam when made by any form of the method of the first aspect of the invention discussed above.

30 The following examples are provided as illustrative of the present invention and are not intended to limit its scope in any way.

## Examples

### Example 1

Resinox CL1916 phenolic resin was mixed with AH2070S acid hardener in a weight ratio of 100 parts resin to 10 parts hardener and mixed by hand using a bowl and a wooden spatula for a period of 2 minutes. A portion of an equivalent mass of 3M Scotchlite K1 soda-lime--borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Acetone was added to maintain a smooth consistency, and a further portion of the microspheres was added, followed by additional acetone. This process of alternating between addition of microspheres and acetone was continued until a mass of microspheres equivalent to the mass of resin had been added. The total volume of acetone added was twenty millilitres. The mixture was then gently stirred for 8 minutes, and subsequently poured into a plaster mould. The specimen was then placed in an oven at 30.9°C for a period of 24 hours. At this stage, the specimen was fully cured.

The resulting syntactic foam has the following properties:

Sphere volume fraction	0.310
Resin volume fraction	0.032
Void fraction	0.658
Sphere Mass Fraction	0.50
Resin mass fraction	0.50
Foam Density (g/cm <sup>3</sup> )	0.077
Tactile Toughness	Medium
Colour	Pearl White

### Example 2

Resinox CL1916 phenolic resin was mixed with AH2070S acid hardener in a weight ratio of 100 parts resin to 10 parts hardener and mixed by hand using a bowl and a wooden spatula for a period of 3 minutes. 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Acetone was added to maintain a smooth consistency, and a further portion of the microspheres was added, followed by additional acetone. This process of alternating between addition of microspheres and acetone was continued until a mass fraction of spheres to resin of 0.60 to 0.40 was obtained. The mixture was stirred to maintain a smooth paste. The total amount of acetone added was twenty five millilitres.

The mixture was then gently stirred for 12 minutes, and subsequently poured into a plaster mould. The specimen was then placed in an oven at 50°C for a period of 24 hours. At this stage, the specimen was fully cured.

The resulting syntactic foam has the following properties:

Sphere volume fraction	0.433
Resin volume fraction	0.030
Void fraction	0.537
Sphere Mass Fraction	0.60
Resin mass fraction	0.40
Foam Density (g/cm <sup>3</sup> )	0.090
Tactile Toughness	Medium
Colour	Pearl White

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Under scanning electron microscopic examination, the syntactic foam had few broken spheres and a good microsphere packing structure as can be seen in figure 1.

10 Example 3

Resinox CL1916 phenolic resin was mixed with AH2070S acid hardener in a weight ratio of 100 parts resin to 10 parts hardener and mixed by hand using a bowl and a wooden spatula for a period of 3 minutes. 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Acetone was added to maintain a smooth consistency, and a further portion of the microspheres was added, followed by additional acetone. This process of alternating between addition of microspheres and addition of acetone was continued until a mass fraction of spheres to resin of 0.70 to 0.30 was obtained. The mixture was stirred to maintain a smooth paste. The total amount of acetone added was twenty millilitres. The mixture was then gently stirred for 10 minutes, and subsequently poured into a plaster mould. The specimen was then placed in an oven at 50°C for a period of 24 hours. At this stage, the specimen was fully cured.

The resulting syntactic foam has the following properties:

Sphere volume fraction	0.411
Resin volume fraction	0.018
Void fraction	0.571
Sphere Mass Fraction	0.70
Resin mass fraction	0.30
Foam Density (g/cm <sup>3</sup> )	0.073
Tactile Toughness	Soft
Colour	Pearl White

Under scanning electron microscopic examination, the syntactic foam had few broken spheres and a good microsphere packing structure as can be  
5 seen in figure 2.

#### Example 4

Resinox CL1916 phenolic resin was mixed with AH2070S acid  
hardener in a weight ratio of 100 parts resin to 10 parts hardener and mixed  
10 by hand using a bowl and a wooden spatula for a period of 3 minutes. 3M  
Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the  
mixture to form a smooth viscous paste. Acetone was added to maintain a  
smooth consistency, and a further portion of the microspheres was added,  
followed by additional acetone. This process of alternating between addition  
15 of microspheres and acetone was continued until a mass fraction of spheres  
to resin of 0.80 to 0.20 was obtained. The mixture was stirred to maintain a  
smooth paste. The total amount of acetone added was twenty five millilitres.  
The mixture was then gently stirred for 10 minutes, and subsequently poured  
into a plaster mould. The specimen was then placed in an oven at 50°C for a  
20 period of 24 hours. At this stage, the specimen was fully cured.

The resulting syntactic foam has the following properties:

Sphere volume fraction	0.335
Resin volume fraction	0.035
Void fraction	0.630
Sphere Mass Fraction	0.80
Resin mass fraction	0.20
Foam Density (g/cm <sup>3</sup> )	0.055
Tactile Toughness	Soft
Colour	Pearl White

Under scanning electron microscopic examination, the syntactic foam had few broken spheres and a good microsphere packing structure as can be seen in figure 3.

#### 5 Example 5

Resinox CL1916 phenolic resin was mixed with AH2070S acid hardener in a weight ratio of 100 parts resin to 10 parts hardener and mixed by hand using a bowl and a wooden spatula for a period of 3 minutes. A portion of an equivalent mass of 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Water was added to maintain a smooth consistency, and a further portion of the microspheres was added, followed by additional water. This process of alternating between addition of microspheres and water was continued until a mass of microspheres equivalent to the mass of resin had been added. The total volume of water added was fifteen millilitres. The mixture was then gently stirred for 15 minutes, and subsequently poured into a plaster mould. The specimen was then placed in an oven at 50°C for a period of 24 hours. At this stage, the specimen was fully cured.

The resulting syntactic foam has the following properties:

Sphere volume fraction	0.472
Resin volume fraction	0.049
Void fraction	0.479
Sphere Mass Fraction	0.50
Resin mass fraction	0.50
Foam Density (g/cm <sup>3</sup> )	0.118
Tactile Toughness	Medium
Colour	Pearl White

Under scanning electron microscopic examination, the syntactic foam had many broken spheres as indicated in figure 4.

#### Example 6

Resinox CL1916 phenolic resin was mixed with AH2070S acid hardener in a weight ratio of 100 parts resin to 10 parts hardener and mixed by hand using a bowl and a wooden spatula for a period of 3 minutes. 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the

mixture to form a smooth viscous paste. Water was added to maintain a smooth consistency, and a further portion of the microspheres were added, followed by additional water. This process of alternating between addition of microspheres and water was continued until a mass fraction of spheres to resin of 0.60 to 0.40 was obtained. The mixture was stirred to maintain a smooth paste. The total amount of water added was fifteen millilitres. The mixture was then gently stirred for 10 minutes, and subsequently poured into a plaster mould. The specimen was then placed in an oven at 50°C for a period of 24 hours. At this stage, the specimen was fully cured.

10 The resulting syntactic foam has the following properties:

Sphere volume fraction	0.507
Resin volume fraction	0.035
Void fraction	0.542
Sphere Mass Fraction	0.60
Resin mass fraction	0.40
Foam Density (g/cm <sup>3</sup> )	0.106
Tactile Toughness	Medium
Colour	Pearl White

Under scanning electron microscopic examination, the syntactic foam had few broken spheres and a good microsphere packing structure as indicated in Figure 5.

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#### Example 7

Synapol 2120 epoxy resin was mixed in Synapol 2120 hardener in a weight ratio of two parts resin to one part hardener and mixed by hand using a bowl and a wooden spatula for a period of three minutes. 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Acetone was added to maintain a smooth consistency, and a further portion of the microspheres was added, followed by additional acetone. This process of alternating between addition of microspheres and addition of acetone was continued until a mass of microspheres equivalent to the mass of resin had been added. The mixture was stirred to maintain a smooth paste. The total amount of acetone added was eighty millilitres. The mixture was then gently stirred for ten minutes, and subsequently poured into a cement mould. The specimen was then left

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to air dry for a period of twenty-three hours. At this stage, the specimen was fully cured.

The resulting syntactic foam has the following properties:

Sphere volume fraction	0.296
Resin volume fraction	0.034
Void fraction	0.670
Sphere Mass Fraction	0.50
Resin mass fraction	0.50
Foam Density (g/cm <sup>3</sup> )	0.0738
Tactile Toughness	Medium
Colour	White

#### 5 Example 8

Synapol 2120 epoxy resin was mixed with Synapol 2120 hardener in a weight ratio of two parts resin to one part hardener and mixed by hand using a bowl and a wooden spatula for a period of three minutes. 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Acetone was added to maintain a smooth consistency, and a further portion of microspheres was added, followed by additional acetone. This process of alternating between addition of microspheres and addition of acetone was continued until a mass fraction of microspheres to resin of 0.60 to 0.40 was obtained. The mixture was stirred to maintain a smooth paste. The total amount of acetone added was ninety millimetres. The mixture was then gently stirred for ten minutes, and subsequently poured into a cement mould. The specimen was then left to air dry for a period of twenty-one hours. At this stage the specimen was fully cured.

20 The resulting syntactic foam has the following properties:

Sphere volume fraction	0.346
Resin volume fraction	0.026
Void fraction	0.628
Sphere Mass Fraction	0.60
Resin mass fraction	0.40
Foam Density (g/cm <sup>3</sup> )	0.072
Tactile Toughness	Medium
Colour	White

### Example 9

Synapol 2120 epoxy resin was mixed with Synapol 2120 hardener in a weight ratio of two parts resin to one part hardener and mixed by hand using a bowl and a wooden spatula for a period of three minutes. 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Acetone was added to maintain a smooth consistency, and a further portion of microspheres was added, followed by additional acetone. This process of alternating between addition of microspheres and addition of acetone was continued until a mass of microspheres equivalent to the mass of resin had been added. The mixture was stirred to maintain a smooth paste. The total amount of acetone added was eighty millilitres. mixture was then gently stirred for ten minutes, and subsequently poured into a plaster mould. The specimen was then left to air dry for a period of twenty-three hours. At this stage, the specimen was fully cured.

The resulting syntactic foam has the following properties:

Sphere volume fraction	0.289
Resin volume fraction	0.033
Void fraction	0.678
Sphere Mass Fraction	0.50
Resin mass fraction	0.50
Foam Density (g/cm <sup>3</sup> )	0.072
Tactile Toughness	Medium
Colour	White

### Example 10

Synapol 2120 epoxy resin was mixed with Synapol 2120 hardener in a weight ratio of two parts resin to one part hardener and mixed by hand using a bowl and a wooden spatula for a period of three minutes. 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Acetone was added to maintain a smooth consistency, and a further portion of microspheres was added, followed by additional acetone. This process of alternating between addition of microspheres and addition of acetone was continued until a mass fraction of spheres to resin of 0.60 to 0.40 was obtained. The mixture was stirred to maintain a smooth paste. The total amount of acetone added was

ninety millilitres. The mixture was then gently stirred for ten minutes, and subsequently poured into a plaster mould. The specimen was then left to air dry for a period of twenty-one hours. At this stage, the specimen was fully cured.

- 5 The resulting syntactic foam has the following properties:

Sphere volume fraction	0.343
Resin volume fraction	0.026
Void fraction	0.631
Sphere Mass Fraction	0.60
Resin mass fraction	0.40
Foam Density (g/cm <sup>3</sup> )	0.071
Tactile Toughness	Soft
Colour	White

#### Example 11

- Synapol 2120 epoxy resin was mixed with Synapol 2120 hardener in a weight ratio of two parts resin to one part hardener and mixed by hand using a bowl and a wooden spatula for a period of three minutes. 3M Scotchlite K1 soda-lime-borosilicate glass spheres were added slowly to the mixture to form a smooth viscous paste. Acetone was added to maintain a smooth consistency, and a further portion of microspheres was added, followed by additional acetone. This process of alternating between addition of microspheres and addition of acetone was continued until a mass fraction of spheres to resin of 0.70 to 0.30 was obtained. The mixture was stirred to maintain a smooth paste. The total amount of acetone added was fifty millilitres. The mixture was then gently stirred for ten minutes, and subsequently poured into a plaster mould. The specimen was then left to air dry for a period of twenty hours. At this stage, the specimen was fully cured.

The resulting syntactic foam has the following properties:

Sphere volume fraction	0.389
Resin volume fraction	0.019
Void fraction	0.592
Sphere Mass Fraction	0.70
Resin mass fraction	0.30
Foam Density (g/cm <sup>3</sup> )	0.069
Tactile Toughness	Soft
Colour	White

Accordingly, the present invention discloses methods of producing syntactic foams which are able to produce foam of low density using techniques which are ideally suited for large scale application. It is to be appreciated that variations and/or additions may be made to the parts and  
5 processes previously defined without departing from the spirit or ambit of the inventions as defined in the following claims.

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THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method of forming a syntactic foam including the steps of:
  - (a) combining a polymer, microspheres and a solvent to form a slurry;
  - (b) thereafter removing a portion of said solvent through a porous wick;
  - 5 and
  - (c) applying conditions which substantially solidify said polymer.
2. The method of claim 1 wherein said polymer and microspheres are combined to form a paste, and thereafter the solvent is combined with said paste to form a slurry.
- 10 3. The method of either claim 1 or 2 wherein said microspheres, said polymer and said solvent are combined together in incremental portions.
4. The method of any preceding claim wherein the slurry is inserted in a mould having a desired physical shape, and the solvent is removed from said slurry while the slurry is in a mould to form a syntactic foam with the desired
- 15 physical shape.
5. The method of claim 4 wherein said mould includes an inner surface in contact with said slurry, and said inner surface forms at least part of the porous wick.
6. The method of any preceding claim wherein said porous wick is
- 20 selected from plaster, clay, ceramic, cement, concrete, finely perforated plastic sheets with absorbent backing, finely perforated metallic sheets with absorbent backing, fine metallic mesh/gauze with absorbent backing, rigid porous foams or sponge-like material.
7. The method of any preceding claim wherein said solvent is water or a
- 25 volatile alcohol or a ketone.
8. The method of any preceding claim wherein said solvent is selected from water, butyl alcohol, isopropyl alcohol, ethyl alcohol, methyl alcohol, acetone or mixtures thereof.
9. The method of claim 8 wherein said solvent is acetone.
- 30 10. The method of any preceding claim wherein said polymer is a resin based compound.
11. The method of claim 10 wherein said resin based compound is selected from amino resins, epoxide resins, phenolic resins, tar acid resins,

urea resins, melamine resins, vinyl resins, styrene resins, alkyd resins, acrylic resins, polyethylene resins, polypropylene resins, petroleum polymer resins, polyamide resins, polycarbonate resins, acetal resins, fluorohydrocarbon resins, polyester resins and polyurethane resins.

- 5 12. The method of claim 10 wherein said polymer is phenolic resin.
13. The method according to claim 10 wherein said polymer is a water soluble polymer.
14. The method according to any one of claims 10 to 13 wherein said polymer further comprises a cross linking agent.
- 10 15. The method according to any preceding claim wherein said microspheres are hollow gas filled spheres.
16. The method according to claim 15 wherein said gas filled spheres are selected from carbon, ceramic, phenolic, epoxy, or melamine-formaldehyde, soda-lime-borosilicate glass.
- 15 17. The method of claim 16 wherein said gas filled spheres are made of soda-lime-borosilicate glass.
18. The method of any preceding claim wherein the surface of said microspheres is pre-coated with a silane coupling agent.
19. The method of any preceding claim wherein the microspheres are less
- 20 than 420 micrometers in diameter.
20. The method of any preceding claim wherein the microspheres and polymer are combined in a weight ratio of between 8:2 and 2:8.
21. The method of claim 20 wherein the weight ratio of microspheres to polymer is between 8:2 and 6:4.
- 25 22. The method of claim 20 wherein the weight ratio of microspheres to polymer is between 8:2 and 7:3.
23. The method of claim 20 wherein the weight ratio of microspheres to polymer is between 1:1 and 2:8.
24. The method of claim 20 wherein the weight ratio of microspheres to
- 30 polymer is between 4:6 and 2:8.
25. The method of claim 20 wherein the weight ratio of microspheres to polymer is between 3:7 and 2:8.

26. The method of any one of claims 20 to 25 wherein the syntactic foam has total void spaces formed from interstitial voids between the microspheres, and microsphere void spaces within the microspheres, wherein the weight ratio of microspheres to polymer forms a syntactic foam with a total interstitial void space greater than the total microsphere void space.
27. The method of any preceding claim wherein the syntactic foam has a density of between 0.05 and 0.5 grams/cm<sup>3</sup>.
28. The method of claim 27 wherein the density of the syntactic foam is between 0.055 grams/cm<sup>3</sup> and 0.15 grams/cm<sup>3</sup>.
29. A syntactic foam when made by a method including the steps of:
- (a) combining a polymer, microspheres and a solvent to form a slurry;
  - (b) thereafter removing a portion of said solvent through a porous wick; and
  - (c) applying conditions which substantially solidify said polymer.
30. A syntactic foam wherein the syntactic foam has total void spaces formed from interstitial voids between the microspheres, and microsphere void spaces within the microspheres, and wherein the total interstitial void space is greater than the total microsphere void space.
31. A syntactic foam formed by a method including the steps of:
- (a) combining a polymer, microspheres and a solvent to form a slurry;
  - (b) thereafter removing a portion of said solvent through a porous wick; and
  - (c) applying conditions which substantially solidify said polymer;
- wherein the syntactic foam has total void spaces formed from interstitial voids between the microspheres, and microsphere void spaces within the microspheres, and wherein the total interstitial void space is greater than the total microsphere void space.
32. A syntactic foam wherein the density of the syntactic foam is between 0.055 grams/cm<sup>3</sup> and 0.15 grams/cm<sup>3</sup>.
33. A method of forming syntactic foam substantially as herein described with reference to any one of the examples.

5 PHILLIPS ORMONDE & FITZPATRICK  
Attorneys for  
ROBERTO DE TOFFOL

David B Fitzpatrick



Figure 1

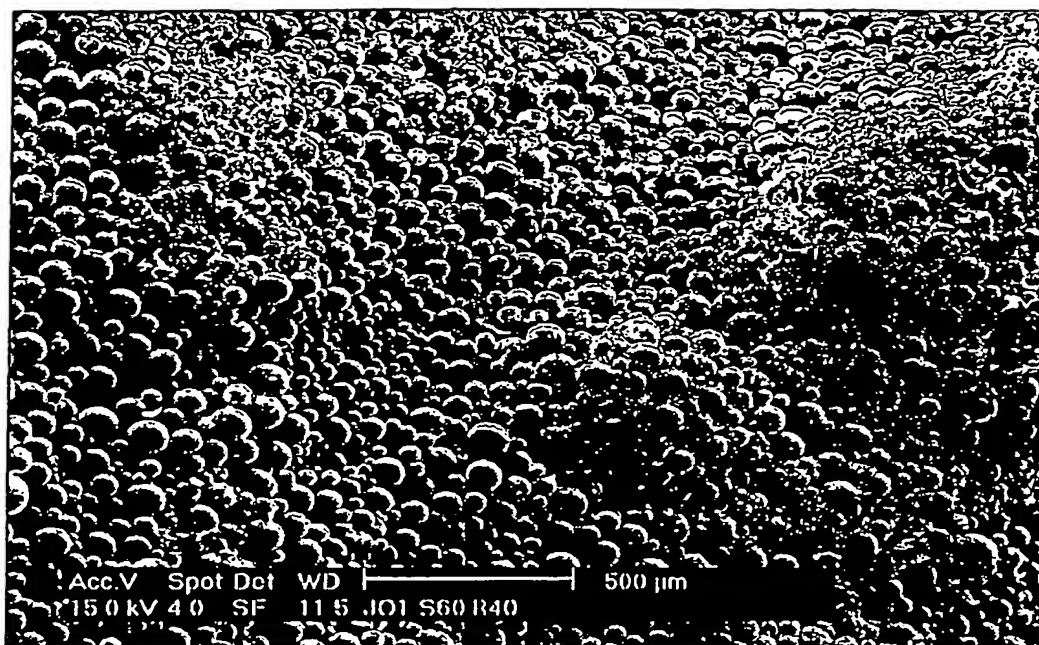


Figure 2



Figure 3

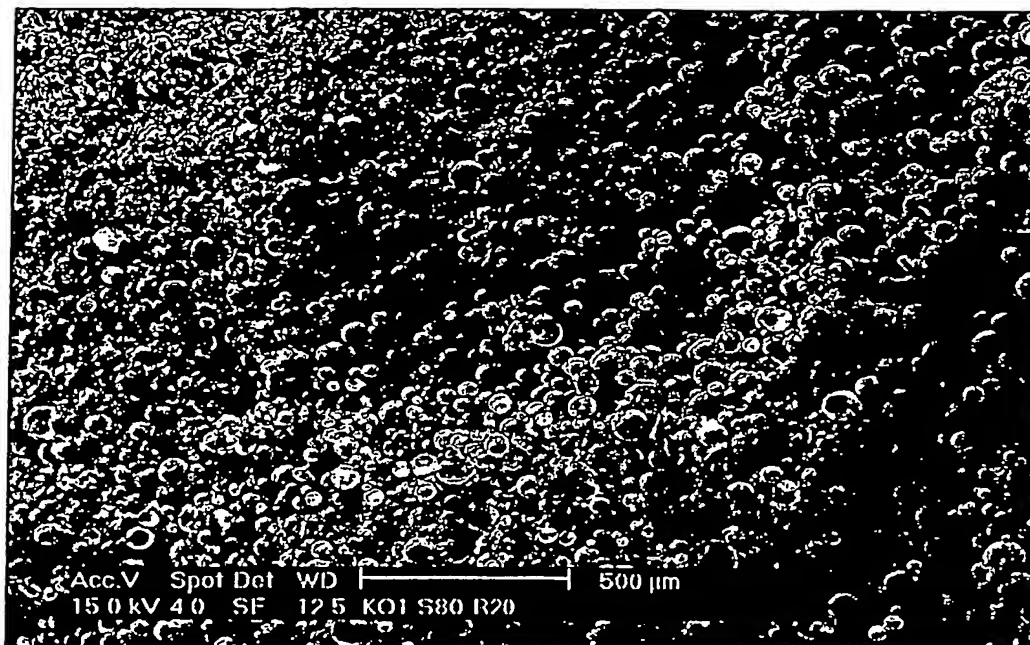


Figure 4

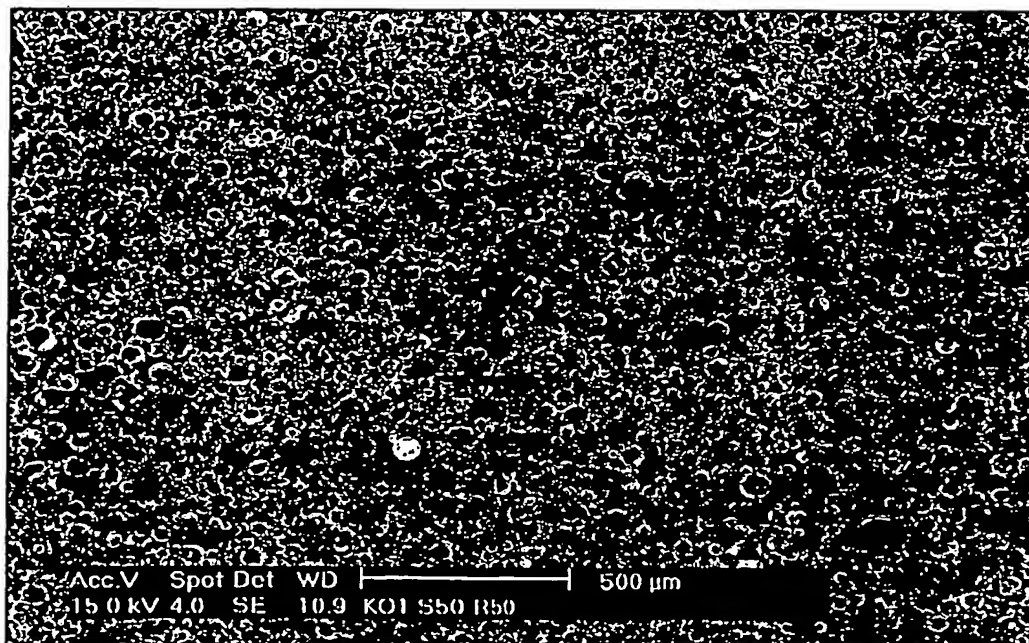
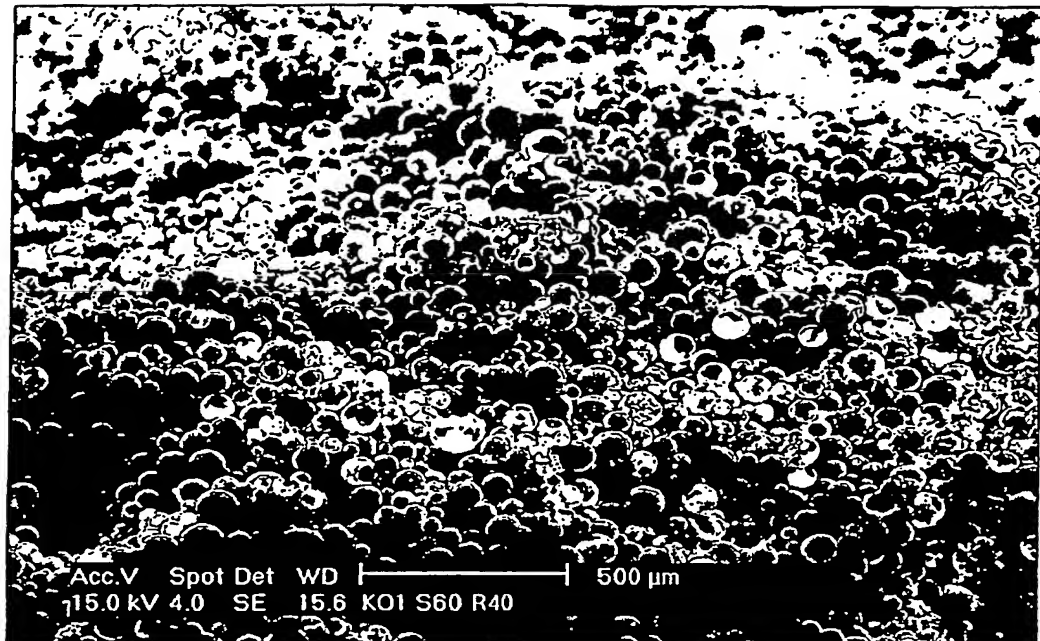


Figure 5



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